

# **Occurrence of Organic Wastewater Contaminants, Pharmaceuticals, and Personal Care Products in Selected Water Supplies, Cape Cod, Massachusetts, June 2004**

By Marc J. Zimmerman

In cooperation with the  
Barnstable County Department of Health and Environment

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# Conversion Factors and Abbreviations

Multiply	By	To obtain
liter (L)	1.057	quart (qt)
liter (L)	0.2642	gallon (gal)
liter (L)	61.02	cubic inch (in <sup>3</sup> )
kilometer (km)	0.6214	mile (mi)
meter (m)	3.281	foot (ft)
meter (m)	1.094	yard (yd)
micrometer ( $\mu\text{m}$ )	0.000003281	foot (ft)

Concentrations of chemical constituents in water are given either in milligrams per liter (mg/L) or micrograms per liter ( $\mu\text{g}/\text{L}$ ).

MDL	method detection limit
MRL	minimum reporting level
NWQL	U.S. Geological Survey National Water Quality Laboratory
OWC	organic wastewater contaminant
PPCP	pharmaceutical and personal care product
SED	suspected endocrine disruptor



# **Occurrence of Organic Wastewater Contaminants, Pharmaceuticals, and Personal Care Products in Selected Water Supplies, Cape Cod, Massachusetts, June 2004**

By Marc J. Zimmerman

## **Abstract**

In June 2004, the U.S. Geological Survey, in cooperation with the Barnstable County Department of Health and Environment, sampled water from 14 wastewater sources and drinking-water supplies on Cape Cod, Massachusetts, for the presence of organic wastewater contaminants, pharmaceuticals, and personal care products. The geographic distribution of sampling locations does not represent the distribution of drinking-water supplies on Cape Cod. The environmental presence of the analyte compounds is mostly unregulated; many of the compounds are suspected of having adverse ecological and human health effects. Of the 85 different organic analyte compounds, 43 were detected, with 13 detected in low concentrations (less than 1 microgram per liter) from drinking-water supplies thought to be affected by wastewater because of previously detected high nitrate concentrations. (Phenol and *d*-limonene, detected in equipment blanks at unacceptably high concentrations, are not included in counts of detections in this report.) Compounds detected in the drinking-water supplies included the solvent, tetrachloroethylene; the analgesic, acetaminophen; the antibiotic, sulfamethoxazole; and the antidepressant, carbamazepine. Nitrate nitrogen, an indicator of wastewater, was detected in water supplies in concentrations ranging from 0.2 to 8.8 milligrams per liter.

## **Introduction**

In the United States, reports of the occurrence and distribution of organic wastewater contaminants (OWCs) including antibiotics, human pharmaceuticals, industrial and household wastewater products (such as pesticides, flame retardants, detergent metabolites, antioxidants, and disinfectants) and hormones in surface water and drinking-water supplies have become commonplace. A U.S. Geological Survey (USGS) national survey (Kolpin and others, 2002) reported the presence of at least one of these compounds in 80 percent of 139 streams sampled in 1999–2000. The effects of some of these compounds may include the feminization of male fish due to the occurrence of estrogen (Jobling and others, 1999; Pickering and Sumpter, 2003). Some of these compounds are classified as “xenobiotics,” molecules foreign to biological systems. Their effects may result in human sperm deoxyribonucleic acid (DNA) damage (Aitken and others, 2004). In the national survey, approximately 40 of the compounds were detected in streams in Massachusetts, some of which are used for water supply; compounds detected include OWCs, steroids, hormones, antibiotics, and pharmaceuticals. About 30 of these compounds were detected during an additional USGS sampling in Massachusetts in 2001 in the raw-water intakes of some public water-supply wells near streams transporting treated wastewater (U.S. Geological Survey, unpub. data, 2001).

## **2      Organic Wastewater Contaminants, Pharmaceuticals, and Personal Care Products in Water Supplies, Cape Cod, MA**

Daughton (2001) identified the need for expanding knowledge and understanding of the occurrence and distribution of pharmaceutical and personal care products (PPCPs) in surface-water sewage and aquatic systems. Until recently, however, even less information was available about the occurrence or distribution of PPCPs in ground water than in surface water. Several studies have considered the transformations of these compounds when they are affected by wastewater and drinking-water treatment processes. These studies indicate that many of these compounds may persist in ground water after treatment (Stackelberg and others, 2004; Cordy and others, 2004; Heberer and others, 2004; Zuehlke and others, 2004; Mansell and Drewes, 2004; Guang-Guo and others, 2004; Snyder and others, 2004; Barnes and others, 2004). Wherever the use of septic systems for wastewater disposal is common and water-supply wells draw water from the same aquifers that support septic systems, the potential exists for these wastewater compounds to enter drinking water supplied by ground water. The risks of exposure to many of these unregulated compounds in drinking water are subjects of current research (Daughton and Ternes, 1999; U.S. Environmental Protection Agency, 1998; National Research Council, 1999, 2001; Daughton, 2004). As knowledge of and concern about the potential effects of OWCs and PPCPs on humans and wildlife increases, State and Federal regulatory agencies may eventually require the application of treatment and disposal technologies for the compounds.

On Cape Cod (Barnstable County, Massachusetts), wastewater is predominately treated by onsite technologies and disposed of through septic systems. Nitrate concentrations above background concentrations have long indicated that wastewater disposal affects public and private ground-water supplies on Cape Cod (Persky, 1986). In Massachusetts, the USGS has participated in national studies (Kolpin and others, 2002; Barnes and others, 2005) assessing the occurrence and distribution of OWCs and PPCPs in surface waters and in water supplies drawn from surface waters, but has not participated in similar studies of water supplies derived from ground water. Thus, in June 2004, the USGS, in cooperation with the Barnstable County Department of Health and Environment, conducted a survey to determine whether concentrations of OWCs and PPCPs were detectable in water samples from selected parts of the aquifers supplying drinking water to residents of Cape Cod, Massachusetts.

This report describes data-collection methods and analytical results for a limited survey of OWCs and PPCPs in Cape Cod drinking water and wastewater. Samples were collected from six wastewater sources and eight drinking-water sources potentially affected by wastewater. Because of the small number of samples and the requirement that samples be collected only where permission was granted, these results do not indicate conditions for all of Cape Cod.

## **Data-Collection Methods**

To determine the extent to which water from public, semipublic, and private drinking-water-supply wells on Cape Cod was affected by OWCs and PPCPs, samples were collected from drinking-water supplies in areas suspected to be affected by wastewater because of previously detected high concentrations of nitrate. To confirm the results of previous investigations, samples were collected from all sampling sites and analyzed for nitrate. Three public supply wells from two towns were sampled, and one semipublic and four private-supply wells were sampled from another town; sampling locations are not included in this report to provide anonymity, a prerequisite for the collection of some samples.

To determine which OWCs and PPCPs may be in treated wastewater, samples were collected from (1) monitoring well 92B at the Barnstable Wastewater Treatment Facility, (2) three monitoring wells approximately 0.4 (C2) and 1.5 km (B1 and B2, colocated, but at different depths) from the Barnstable facility, (3) a standard septic-tank leachfield (Massachusetts Title 5) system, and (4) a recirculating sand filter system. The two latter systems were at the Massachusetts Alternative Onsite Septic System Test Center on the Massachusetts Military Reservation in Falmouth. Minimizing the amount of nitrogen returned to the aquifer is an important consideration in the design and operation of these wastewater-treatment facilities, but they are not expected to remove OWCs and PPCPs. Because this report does not include information on the volume of wastewater processed, however, the nitrate concentrations detected do not indicate the effectiveness of the test systems in removing nitrogen from wastewater.

A peristaltic pump was used to collect samples from monitoring wells after withdrawing at least three well-casing volumes of water. Teflon bailers were used to collect samples manually from holding tanks at the onsite test facility. Drinking-water samples were collected from spigots or sinks at public-water supplies, private homes, and one semipublic water supply (a restaurant). None of the water supplies had water softeners or other treatment systems. Sample collection and processing followed the standard USGS protocols (Wilde and others, 2003). Water samples to be analyzed for organic compounds were collected in 3-L Teflon bottles that were washed with a 0.2 percent solution of nonphosphate laboratory-grade detergent, rinsed with copious amounts of laboratory-grade deionized water, then rinsed with pesticide-grade methanol. The 3-L Teflon bottles were rinsed onsite three times with sample water before samples were collected. Samples were processed by filtration through 0.7- $\mu\text{m}$  glass microfiber filters into precleaned, baked 1-L amber glass bottles that were then stored on ice before and during shipment to the USGS National Water Quality Laboratory (NWQL) in Denver, Colorado, for analysis. These samples were collected on June 1 and 2, 2004, and were shipped overnight to the laboratory on June 4, 2004. The OWCs were analyzed by gas chromatography-mass spectrometry (Zaugg and others, 2001), and PPCPs were analyzed by high-pressure liquid chromatography-mass spectrometry (Cahill and others, 2004).

Six environmental samples were obtained from wastewater sources, and 10 environmental samples, including replicate samples, were obtained from drinking-water supplies. Replicate samples were collected from two of the private drinking-water wells. On each sampling day, one equipment-blank sample was collected by filling sample bottles with volatile organic compound (VOC) grade blank water and filtering in the same manner as the environmental samples. At each sampling site, an additional unfiltered water sample was collected in polyethylene bottles and delivered to the Barnstable County Health Laboratory for nitrate analysis; these samples were delivered to the laboratory on the day of collection.

## Occurrence of Wastewater Contaminants, Pharmaceuticals, and Personal Care Products

In the samples collected, 43 organic OWC and PPCP compounds were detected (tables 1 and 2, at back of report); caffeine and cotinine, listed in both tables, were not counted twice. (Phenol and *d*-limonene, detected in equipment blanks at unacceptably high concentrations, are not included in counts of detections in this report.) Nine of the OWCs detected were suspected endocrine disruptors (SEDs). The OWC minimum reporting levels (MRLs) ranged from 0.5 to 5.0  $\mu\text{g/L}$ ; 84 of the concentrations detected were reported as estimated values less than the minimum reporting levels (MRLs). The PPCP analytical method, a research method at the time of the study, had provisional method detection levels (MDLs) ranging from 0.002 to 0.072  $\mu\text{g/L}$  (J.D. Cahill, Chemist, U.S. Geological Survey, written commun., 2005).

The NWQL adds surrogate compounds (compounds that are expected to perform similarly to the compounds being analyzed in a laboratory method) to all environmental and quality-control samples to monitor compound recovery and potential environmental effects (Pirkey and Glodt, 1998). Surrogate recoveries (not included in table 1) for the OWC method ranged from 85 to 130 percent. The surrogate recoveries (not included in table 2) for the PPCP method were 101 and 102 percent.

Three compounds, *d*-limonene, phenol, and acetaminophen, were detected in equipment blank samples. Phenol concentrations (E0.370 and 0.54  $\mu\text{g/L}$ ) in the blank samples were greater than some of the phenol concentrations detected in environmental samples and were a substantial fraction of the phenol concentrations of other samples. Acetaminophen and *d*-limonene were detected at estimated concentrations of E0.010 and 0.110  $\mu\text{g/L}$ , respectively, in the equipment-blank sample collected on June 2. Of the seven environmental samples collected that day and analyzed in the same batch of samples at the NWQL, two samples had acetaminophen concentrations that were less than the concentration in the equipment-blank sample, and a third environmental sample had a concentration of 0.023  $\mu\text{g/L}$ , slightly more than twice the concentration in the equipment-blank sample; the concentration of *d*-limonene in the environmental sample was approximately three times the concentration in the equipment-blank sample. For these reasons, the concentrations of phenol, acetaminophen, and *d*-limonene in environmental samples are dubious, and the samples should be considered contaminated with respect to these compounds.

## **4      Organic Wastewater Contaminants, Pharmaceuticals, and Personal Care Products in Water Supplies, Cape Cod, MA**

Of the compounds detected, 43 were in samples collected from monitoring wells at or near the wastewater-treatment plant (92B, C2, B1, B2) or the onsite test area. The compounds include petrochemicals frequently associated with gasoline (naphthalene and related compounds), flame retardants (bisphenol A, tri(dichloroisopropyl) phosphate, tri(2-butoxyethyl) phosphate, and tri(2-chloroethyl) phosphate), and detergent metabolites (octylphenols and nonylphenols) (table 1).

Thirteen compounds were detected in samples from drinking-water supplies, and these compounds were detected less frequently than those detected in samples from the monitoring wells or from the onsite test area. Fewer OWCs and PPCPs were detected in the public-water supplies than in the private or semipublic water supplies. Three analytes were detected in samples from the public water supplies: tetrachloroethylene (a solvent), phenol (a disinfectant; as discussed, phenol detections may be due to sample contamination, not occurrence in the water supply), and sulfamethoxazole (an antibiotic); no contaminants were detected in the third public-water supply. Unlike the public-supply wells, samples from one private well (private well 1, tables 1 and 2) and from the semipublic water-supply well contained detectable concentrations of plasticizers (ethyl and phenyl phosphates) and flame retardant (methyl and butyl phosphates) compounds. None of the contaminant concentrations in the drinking-water supplies exceeded the MRL.

In water-supply wells, the highest nitrate concentration was 8.8 mg/L in a sample from a private well, and the lowest concentration was 0.2 mg/L in a sample from a public water-supply well (table 1). The highest nitrate concentration (28 mg/L) was found in the sample from the recirculating sand filter holding tank; a sample from one of the wastewater-discharge monitoring wells (C2) had a nitrate concentration less than the MRL of 0.1 mg/L.

## **Summary**

In cooperation with the Barnstable County Department of Health and Environment, the U.S. Geological Survey collected samples from wastewater sources and drinking-water supplies on Cape Cod to determine whether organic wastewater contaminants, pharmaceuticals, and personal care products were in public, semipublic, and private water supplies. Many of the target analytes are suspected of being potentially harmful to humans and wildlife. Forty-three of the compounds were detected in the wastewater sources and 13 in the water supplies. In uncontaminated samples from the wastewater sources, concentrations of detected organic wastewater contaminants ranged from 0.0190 to 3.4 µg/L, and concentrations of detected pharmaceutical and personal care compounds ranged from 0.0036 to 6.4 µg/L; in samples from the water supplies, concentrations of detected organic wastewater contaminants ranged from 0.0120 to 0.480 µg/L, and concentrations of pharmaceutical and personal care compounds ranged from 0.0037 to 0.0576 µg/L. Nitrate concentrations in all samples ranged from less than 0.1 to 28 mg/L. Three of the compounds were detected in equipment-blank samples. The geographic distribution of the sampling sites is not representative of Cape Cod; therefore, sampling results are not indicative of conditions for all of Cape Cod.

## **Acknowledgments**

George Heufelder, Director, Barnstable County Department of Health and Environment, enthusiastically supported this project by locating public-water suppliers and private individuals who gave permission to collect samples. Kimberly W. Campo, Hydrologic Technician, U.S. Geological Survey, managed the collection, field processing, and shipping of the samples. U.S. Geological Survey colleague reviewers, Leslie DeSimone of the Massachusetts-Rhode Island Water Science Center and Patrick Phillips of the New York Water Science Center, made valuable comments that substantially improved this report's technical presentation. Steven Zaugg and Jeffrey Cahill, chemists at the U.S. Geological Survey National Water Quality Laboratory, provided valuable explanations about their quality control procedures.

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## **Tables 1 and 2**

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**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.

[Possible use or source: Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, minimum reporting level; NS, not sampled; SED, suspected endocrine disruptor; <, actual value is less than value shown; --, not applicable]

**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

[Possible use or source: Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study; E, estimated value; MRL, minimum reporting level; NS, not sampled; SED, suspected endocrine disruptor; <, actual value is less than value shown; -, not applicable]

Analyte	Possible use or source	SED	MRL	Wastewater sources					Recirculating sand filter	Septic tank test system	
				Quality-control samples	Blank water	Blank water	92B	C2	B1	B2	
Carbaryl	herbicide	Yes	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Carbazole	insecticide, dyes, explosives, lubricants	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Chlorpyrifos	insecticide	Yes	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Cholesterol	fecal indicator, plant sterol	No	2	<2.0	<2.0	<2.0	E1.00	<2.0	<2.0	E3.40	E.900
Cotinine	nicotine metabolite	No	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	E.440	<1.0
Diazinon	insecticide	Yes	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Dichlorvos	insecticide	Yes	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Diethoxyxonylphenol (total)	nonionic detergent metabolite	Yes	5	<5.0	<5.0	<5.0	<5.0	<5.0	<5.0	E1.50	<5.0
Diethoxyoctylphenol	nonionic detergent metabolite	Yes	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
d-Limonene	antimicrobial, antiviral, aerosol fragrance	No	.5	<.5	E.110	<.5	<.5	<.5	<.5	<.5	<.5
Fluoranthene	coal tar, asphalt	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Hexahydrohexamethylcyclpentabenzopyran (HHCB)	musk fragrance	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	E.310	E.170
Indole	pesticide ingredient, fragrance in coffee	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	E.0730	<.5
Isoborneol	fragrance	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Isophorone	solvent for lacquer, plastic, oil, resin	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Isopropylbenzene (cumene)	fuels, paint thinner	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Isoquinoline	flavors and fragrances	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Menthol	cigarettes, cough drops, mouthwash	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	E.350	<.5
Metalaxyl	general-use pesticide	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Methyl salicylate	liniment, food, beverage	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Metolachlor	herbicide	No	.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5	<.5
Monoethoxyoctylphenol	nonionic detergent metabolite	Yes	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
N,N-diethyl-meta-toluamide (DEET)	mosquito repellent	No	.5	<.5	E.110	E.140	E.220	E.150	.63	E.110	
Naphthalene	fumigant, moth repellent, gasoline	No	.5	<.5	E.0330	E.0300	E.0580	<.5	<.5	<.5	<.5
Para-Nonylphenol (total)	nonionic detergent metabolite	Yes	5	<5.0	E.2.70	E1.80	<5.0	E1.10	E2.40	<5.0	

**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

[Possible use or source: Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, minimum reporting level; NS, not sampled; SED, suspected endocrine disruptor; <, actual value is less than value shown; --, not applicable]

Analyte	Possible use or source	Wastewater sources							
		Quality-control samples			Monitoring wells				
		SED	MRL	Blank water	Blank water	C2	B1	B2	Recirculating sand filter
<i>p</i> -Cresol	wood preservative	No	1	<1.0	<1.0	<1.0	<1.0	<1.0	<1.0
Pentachlorophenol	herbicide, fungicide, wood preservative	Yes	2	<2.0	<2.0	<2.0	<2.0	<2.0	<2.0
Phenanthrene	explosives, tar, diesel fuel, crude oil	No	.5	<.5	<.5	<.5	<.5	<.5	<.5
Phenol	disinfectant	No	.5	E.370	0.54	0.98	<.5	E.330	<.5
Prometon	herbicide	No	.5	<.5	<.5	<.5	<.5	<.5	<.5
Pyrene	coal tar, asphalt	No	.5	<.5	<.5	<.5	<.5	<.5	<.5
Tetrachloroethylene	solvent, degreaser, antihelmintic	No	.5	<.5	<.5	E.0640	E.0490	E.0380	<.5
Tri(2-butoxyethyl) phosphate	flame retardant	No	.5	<.5	<.5	E.410	<.5	<.5	<.5
Tri(2-chloroethyl) phosphate	plasticizer, flame retardant	Yes	.5	<.5	<.5	E.190	E.220	E.240	E.0810
Tri(dichloroisopropyl) phosphate	flame retardant	Yes	.5	<.5	<.5	E.370	E.370	E.220	<.5
Tributyl phosphate	flame retardant, antifoaming agent	No	.5	<.5	<.5	E.200	E.190	E.200	<.5
Triclosan	disinfectant, antimicrobial	Yes	1	<1.0	<1.0	E.170	E.180	<1.0	E.280
Triethyl citrate (ethyl citrate)	cosmetics, pharmaceuticals	No	.5	<.5	<.5	<.5	<.5	<.5	E.260
Triphenyl phosphate	plasticizer, resin, wax	No	.5	<.5	<.5	E.0600	<.5	<.5	<.5

**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

(Possible use or source: Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, method reporting level; NS, not sampled; SED, suspected endocrine disruptor;  $\leq$ , actual value is less than value shown; --, not applicable)

**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

[Possible use or source. Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRI, method reporting level; NS, not sampled; SED, suspected endocrine disruptor; <, actual value is less than value shown; --, not applicable.]

**Table 1.** Nitrate and organic wastewater-contaminant concentrations in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

[Possible use or source: Listings are not exhaustive. All concentrations in micrograms per liter except nitrate, which is in milligrams per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, method reporting level; NS, not sampled; SED, suspected endocrine disruptor; <, actual value is less than value shown; --, not applicable]

**Table 2.** Concentrations of pharmaceutical and personal care products in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.

All concentrations are in micrograms per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, method detection limit; MDL, minimum reporting level; ND, not determined; \*, not detected, minimum reporting level not determined; <, actual value is less than value shown; --, not applicable

**Table 2.** Concentrations of pharmaceutical and personal care products in samples collected on Cape Cod, Massachusetts, June 1–2, 2004.—Continued

[All concentrations are in micrograms per liter. Shading indicates analytes that were detected in this study. E, estimated value; MRL, method detection limit; MRL, minimum reporting level; ND, not determined; \*, not detected, minimum reporting level not determined; <, actual value is less than value shown; -, not applicable]